

**WIDE RANGE ULTRAVIOLET ABSORBER**

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**Publication date:** 1999-10-12  
**Inventor:** SAGAWA SEIJI  
**Applicant:** KYODO CHEM CO LTD  
**Classification:**  
**- International:** C07D251/34; C07D401/14; C07D487/04; C07D519/00;  
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C07D519/00; C09K3/00; (IPC1-7): C09K3/00; C07D251/34;  
C07D401/14; C07D487/04; C07D519/00  
**- european:**  
**Application number:** JP19980121594 19980326  
**Priority number(s):** JP19980121594 19980326

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**Abstract of JP11279523**

**PROBLEM TO BE SOLVED:** To obtain a compd. which absorbs wide range ultraviolet rays by forming a tris (epoxypropyl) isocyanurate compd. **SOLUTION:** This isocyanurate compd., which is an absorber for wide range ultraviolet rays of 250-470 nm and/or an ultraviolet absorber for ultrathin films (e.g. coating films), is represented by formula I and is prepd. by adding tris (epoxypropyl) isocyanurate and an equimolar amt. of 2,4-dihydroxybenzophenone to a solvent (e.g. xylene), thermally refluxing the mixture for a certain time, adding an equimolar amt. of p-methoxycinnamic acid followed by thermal refluxing, adding 2-(2,4-dihydroxyphenyl)-5-chlorobenzotriazole, etc., followed by thermal reaction for a certain time, distilling off the solvent under vacuum, and adding an isopropyl alcohol-acetone mixture to precipitate the resultant product. In formula I, R1, R2, and R3 are each a group represented by formula II, III, or IV; and R4 is H or a halogen.

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(11) 特許出願公開番号

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1 3 3

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薬品株式会社内

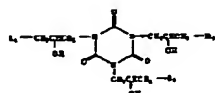
(54) 【発明の名称】 広域紫外線の吸収剤

(57) 【要約】

【課題】 広域紫外線の吸収剤、及び、又は塗膜のような超薄膜用の紫外線吸収剤を提供する。

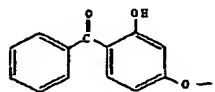
【解決手段】 下記一般式、化1で示されるイソシアヌレート化合物を添加する。

【化1】

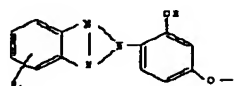


ここで、R1、R2、R3は下記のグループ(化2、化3、化4、化5、化6、化7)から選ばれる同種、又は異種の基を意味する。

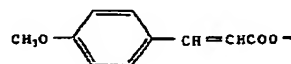
【化2】



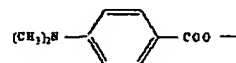
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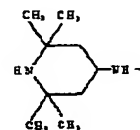
【化4】



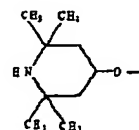
【化5】



【化6】



【化7】



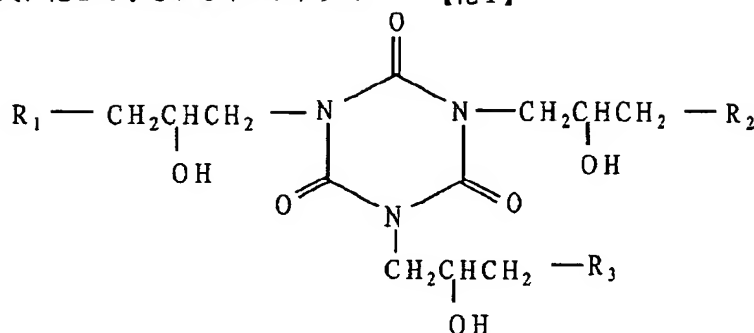
但し、R4は水素、又はハロゲンを意味する。

【特許請求の範囲】

【請求項1】 下記一般式、化1で示されるイソシアヌ\*

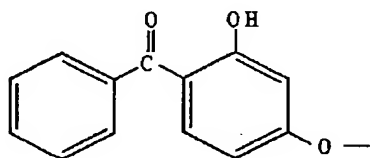
\*レート化合物。

【化 1】

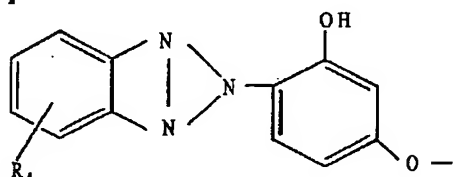


但し、R 1、R 2、R 3は下記のグループ（化2、化3、化4、化5、化6、化7）から選ばれる同種、又は異種の基を意味する。

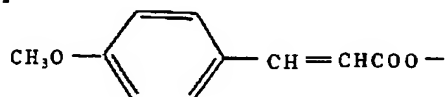
【化2】



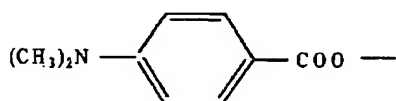
【化3】



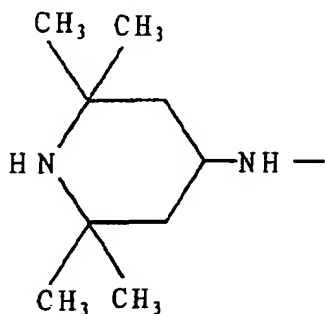
【化4】



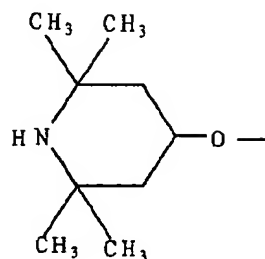
【化5】



【化 6】



【化 7】



20

但し、R<sup>4</sup>は水素、又はハロゲンを意味する。

## 【発明の詳細な説明】

【0 0 0 1】

【発明の属する技術分野】本発明のイソシアヌレート化合物は250 nmから470 nmに至る広域紫外線の吸収剤を、及び又は塗膜のような超薄膜用の紫外線吸収剤の提供にある。

【 0 0 0 2 】

【従来の技術】従来、広域紫外線による基質の劣化を防止するためには、ベンゾトリアゾール系紫外線吸収剤、ベンゾフェノン系紫外線吸収剤、置換桂皮酸エステル類、パラアミノ安息香酸エステル類、ヒンダードアミン系のHALSなど、既往の化合物を適当に組み合わせて使用することによって対応してきた。

【0003】

【発明が解決しようとする課題】 従来技術による場合には、それぞれの化合物の分子量が小さいため、プラスチック製品の加工段階で揮散したり、あるいは使用段階で、ブリード、蒸散、溶出などにより効果が減少する欠点があった。特に塗料を塗布した場合のような超薄膜の場合には、この傾向は一層顕著であり、深刻な問題である。本発明はこの問題を解決しようとするものである。

【 0 0 0 4 】

【課題を解決するための手段】前項の課題を解決することを目的に、従来は個々の紫外線吸収剤に長鎖アルキル基や、ポリオキシアルキレン基などを導入して、分子量を大きくする試みがなされたが、これらの方法ではポリマーに対する相溶性の低下、あるいはモル吸光度の低下などの問題が残り、満足するに至っていない。

50 【0005】本発明者は、ポリマーに対する相溶性を低

下することなく、またモル吸光度の著しい低下を招くことなく、各成分の分子量を同時に上げるためにイソシアヌレート環を基幹とし、これに各成分を化学結合させるという手段を見出し、本発明を完成するに至った。

【0006】すなわち、本発明の化合物は下記一般式、化1で示されるイソシアヌレート化合物である。

【0007】

【化1】

【0008】但し、R1, R2, R3は下記のグループ(化2, 化3, 化4, 化5, 化6, 化7)から選ばれる同種、又は異種の基を意味する。

【0009】

【化2】

【0010】

【化3】

【0011】

\*【化4】

【0012】

【化5】

【0013】

【化6】

【0014】

【化7】

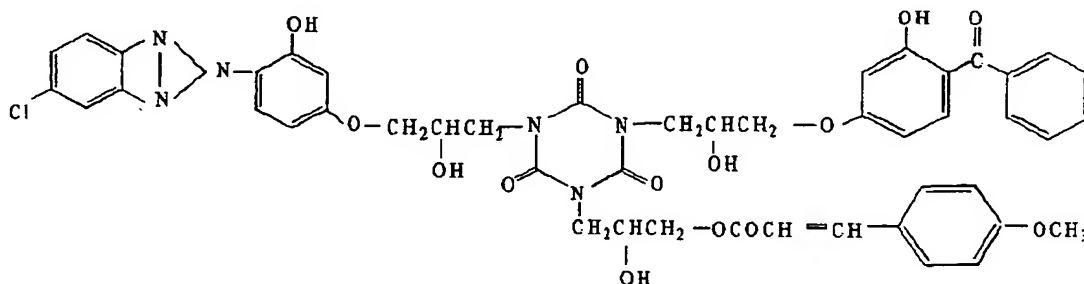
【0015】但し、R4は水素、又はハロゲンを意味する。

【0016】上記一般式に含まれる化合物の例としては、次のような化8, 化9, 化10, 化11, 化12, 化13が挙げられるが、これらに限られるものではない。

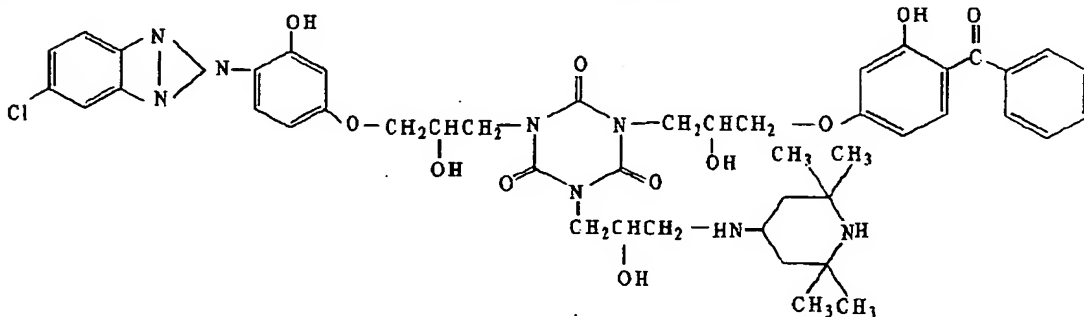
【0017】

【化8】

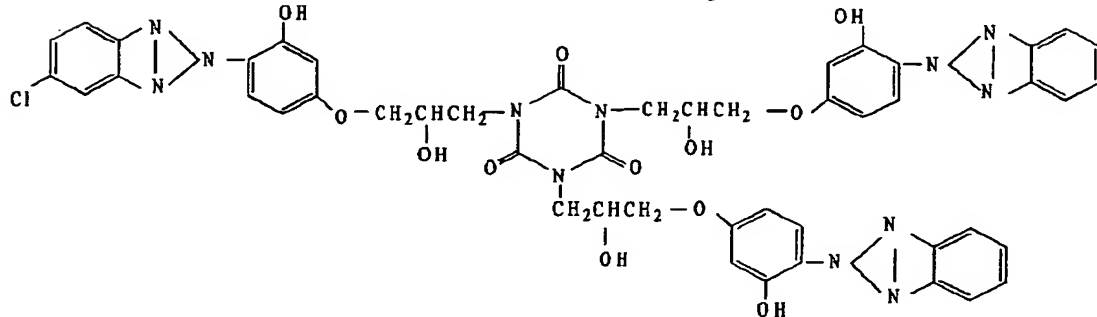
\*



【0018】

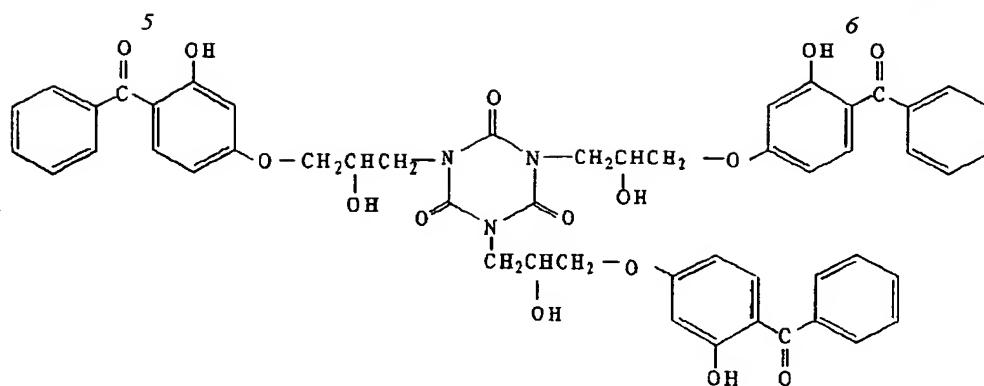


【0019】



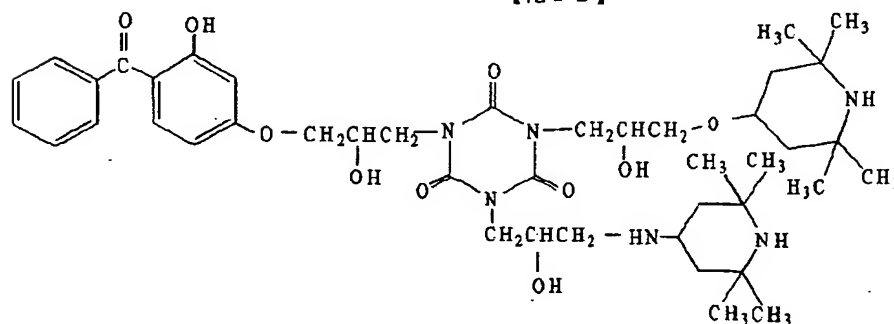
【0020】

【化11】



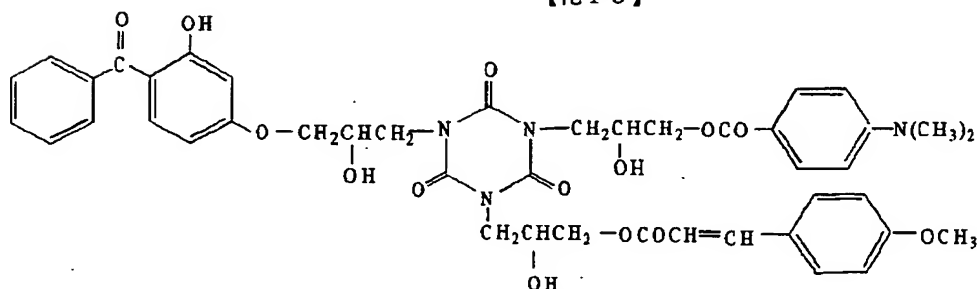
【0021】

【化12】



【0022】

【化13】



【0023】

【実施例】以下、本発明の化合物を実施例で説明する。

【実施例1】化8の合成

300ml容フラスコにキシレン50.0g、2,4-ジヒドロキシベンゾフェノン（共同薬品製）15.0g（0.07モル）、およびトリス（エポキシプロピル）イソシアヌレート（日産化学製）21.5g（0.07モル）を仕込み、140℃で2時間撹拌した。ここでパラメトキシ桂皮酸（和光純薬）12.5g（0.07モル）を添加し、更に2時間撹拌後、2-(2',4'-ジヒドロキシフェニル)-5-クロロベンゾトリアゾール（共同薬品製）18.3g（0.07モル）を添加し、更に5時間加熱撹拌した。その後、減圧下キシレンを溜去し、淡黄褐色透明液を得た。これにイソプロピルアルコールとアセトンの9:1混合溶媒を加えて室温まで冷却し、析出結晶を濾過、乾燥して、融点93~97℃の淡黄褐色粉体64g（収率：理論の96.2%）を得た。紫外吸収領域は250nm~470nmを示した。

30 【0024】【実施例2】化9の合成

実施例1で使用したパラメトキシ桂皮酸の代わりに4-アミノ-2,2,6,6-テトラメチルピペリジン（Huls社製）11g（0.07モル）を使用して、実施例1と同様な操作を行って、融点111~115℃の黄褐色粉体62g（収率：理論の95.4%）を得た。紫外吸収領域は250nm~475nmを示した。

【0025】【実施例3】化13の合成

実施例1の2-(2',4'-ジヒドロキシフェニル)-5-クロロベンゾトリアゾールの代わりにパラジメチルアミノ安息香酸（和光純薬）11.6g（0.07モル）を使用して、実施例1と同様な操作を行って、融点79~84℃の淡褐色粉体53.8g（収率：理論の90.0%）を得た。紫外吸収領域は250nm~360nmを示した。

【0026】

【発明の効果】本発明は広域紫外線の吸収剤、及び又は塗膜のような超薄膜用の紫外線吸収剤を提供するものである。

(5)

特開平 1 1 - 2 7 9 5 2 3

フロントページの続き

(51) Int. Cl. <sup>6</sup>  
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識別記号  
3 1 1

F I  
C 0 7 D 519/00

3 1 1

## PATENT ABSTRACTS OF JAPAN

(11)Publication number : 11-279523

(43)Date of publication of application : 12.10.1999

(51)Int.Cl. C09K 3/00  
C07D251/34  
C07D401/14  
C07D487/04  
C07D519/00

(21)Application number : 10-121594

(71)Applicant : KYODO CHEM CO LTD

(22)Date of filing : 26.03.1998

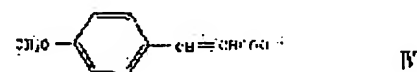
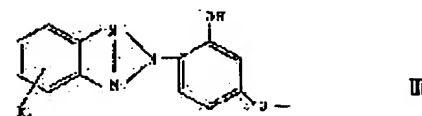
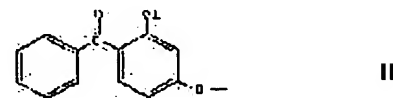
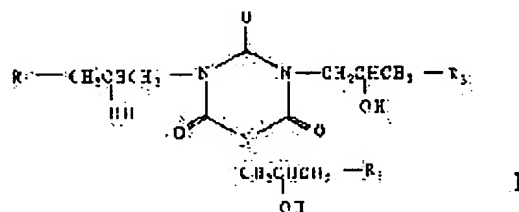
(72)Inventor : SAGAWA SEIJI

## (54) WIDE RANGE ULTRAVIOLET ABSORBER

## (57)Abstract:

PROBLEM TO BE SOLVED: To obtain a compd. which absorbs wide range ultraviolet rays by forming a tris (epoxypropyl) isocyanurate compd.

SOLUTION: This isocyanurate compd., which is an absorber for wide range ultraviolet rays of 250-470 nm and/or an ultraviolet absorber for ultrathin films (e.g. coating films), is represented by formula I and is prepd. by adding tris (epoxypropyl) isocyanurate and an equimolar amt. of 2,4-dihydroxybenzophenone to a solvent (e.g. xylene), thermally refluxing the mixture for a certain time, adding an equimolar amt. of p-methoxycinnamic acid followed by thermal refluxing, adding 2-(2,4-dihydroxyphenyl)-5-chlorobenzotriazole, etc., followed by thermal reaction for a certain time, distilling off the solvent under vacuum, and adding an isopropyl alcohol-acetone mixture to precipitate the resultant product. In formula I, R1, R2, and R3 are each a group represented by formula II, III, or IV; and R4 is H or a halogen.



## LEGAL STATUS

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[Date of sending the examiner's decision of rejection]

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[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

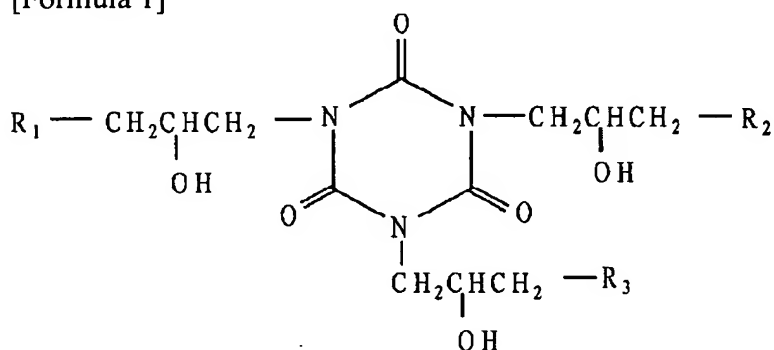
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**[Claim(s)]**

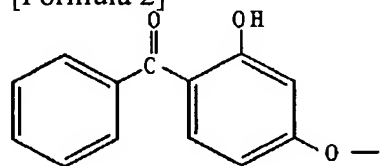
[Claim 1] The following general formula, the isocyanurate compound shown by \*\* 1.

[Formula 1]

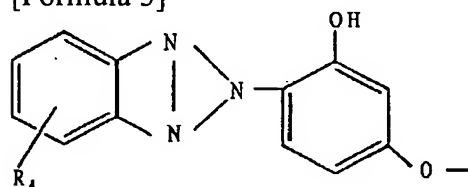


However, R1, R2, and R3 mean the congener chosen from the following group (\*\* 2, \*\* 3, \*\* 4, \*\* 5, \*\* 6, \*\* 7), or a radical of a different kind.

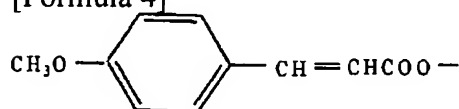
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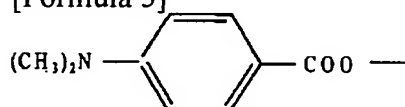
[Formula 3]



[Formula 4]

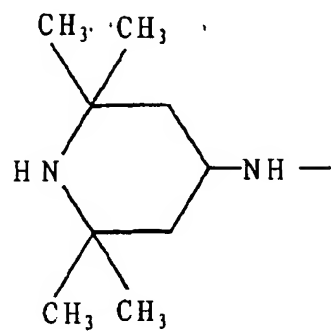


[Formula 5]

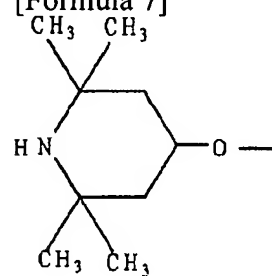


[Formula 6]





[Formula 7]



However, R<sub>4</sub> means hydrogen or a halogen.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention] About the absorbent of broader-based ultraviolet rays from 250nm to 470nm, the isocyanurate compound of this invention reaches or is in offer of an ultraviolet ray absorbent for super-thin films like a paint film.

[0002]

[Description of the Prior Art] In order to prevent degradation of the substrate by broader-based ultraviolet rays conventionally, HALS of a benzotriazol system ultraviolet ray absorbent, a benzophenone system ultraviolet ray absorbent, permutation cinnamic acid ester, p-aminobenzoic-acid ester, and a hindered amine system etc. has corresponded by using it, combining a past compound suitably.

[0003]

[Problem(s) to be Solved by the Invention] Since the molecular weight of each compound was small when based on the conventional technique, it vaporized in the processing phase of a plastic, or there was a fault in which effectiveness decreases according to bleeding, evapotranspiration, elution, etc. in an activity phase. In [ like / at the time of applying especially a coating ] a super-thin film, this inclination is much more remarkable and is a serious problem. This invention tends to solve this problem.

[0004]

[Means for Solving the Problem] Although the attempt which introduces a long-chain alkyl group, a polyoxyalkylene group, etc. into each ultraviolet ray absorbent conventionally, and enlarges molecular weight was made for the purpose of solving the technical problem of the preceding clause, problems, such as lowering of compatibility to a polymer or lowering of a mol absorbance, remain, and it has come to be satisfied with these approaches.

[0005] Without [ without it falls the compatibility over a polymer, and ] causing remarkable lowering of a mol absorbance, this invention person came to complete a header and this invention for a means to carry out the chemical bond of each component to this based on an isocyanurate ring, in order to raise the molecular weight of each component simultaneously.

[0006] That is, the compounds of this invention are the following general formula and an isocyanurate compound shown by \*\* 1.

[0007]

[Formula 1]

[0008] However, R1, R2, and R3 mean the congener chosen from the following group (\*\* 2, \*\* 3, \*\* 4, \*\* 5, \*\* 6, \*\* 7), or a radical of a different kind.

[0009]

[Formula 2]

[0010]

[Formula 3]

[0011]

[Formula 4]

[0012]

[Formula 5]

[0013]

[Formula 6]

[0014]

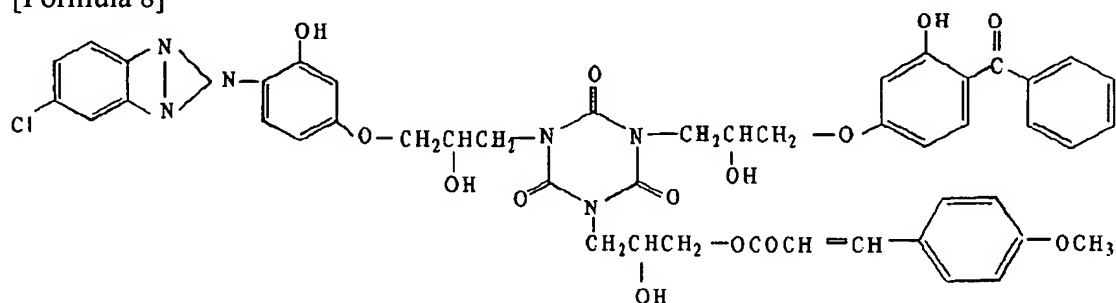
[Formula 7]

[0015] However, R4 means hydrogen or a halogen.

[0016] As an example of the compound contained in the above-mentioned general formula, although following \*\* 8, \*\* 9, \*\* 10, \*\* 11, \*\* 12, and \*\* 13 are mentioned, it is not restricted to these.

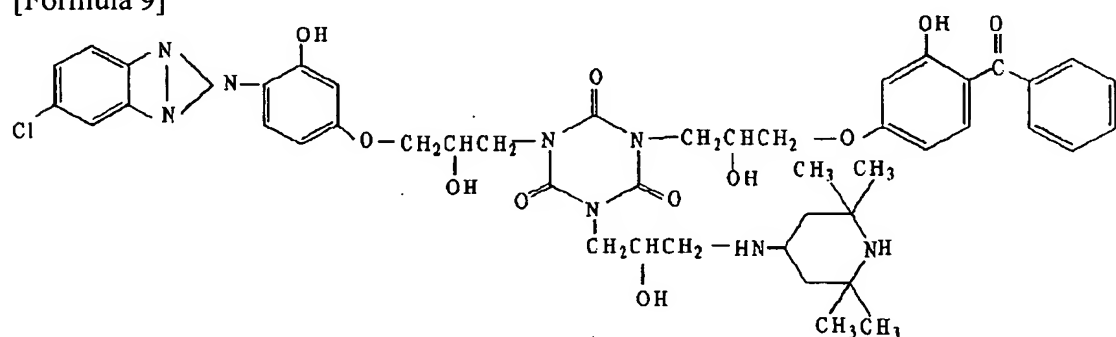
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[Formula 8]



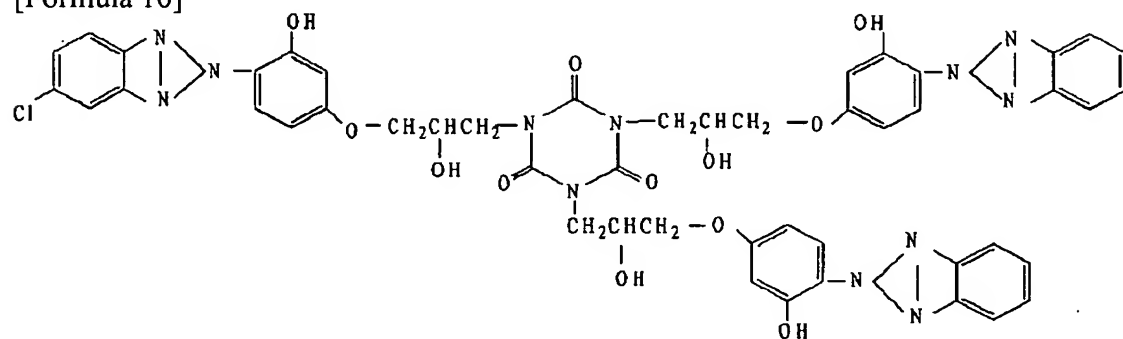
[0018]

[Formula 9]



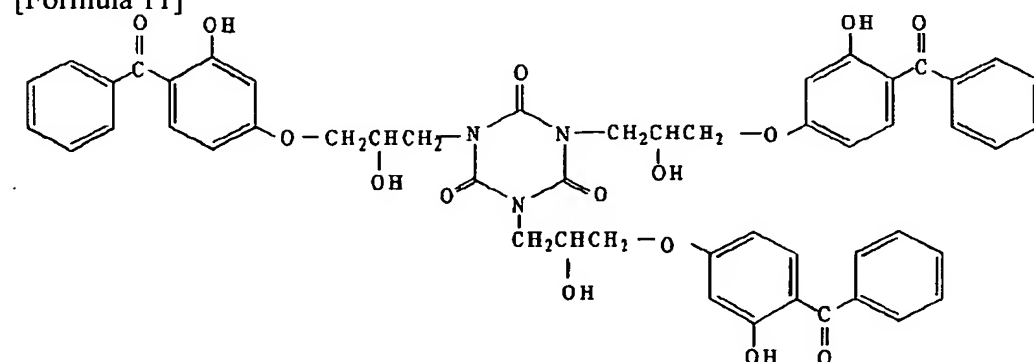
[0019]

[Formula 10]



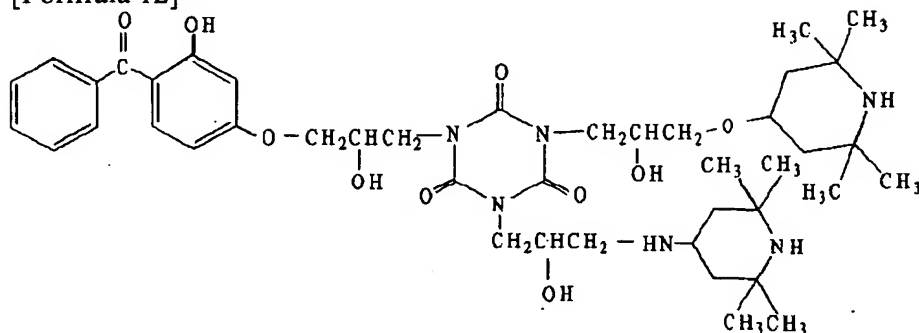
[0020]

[Formula 11]



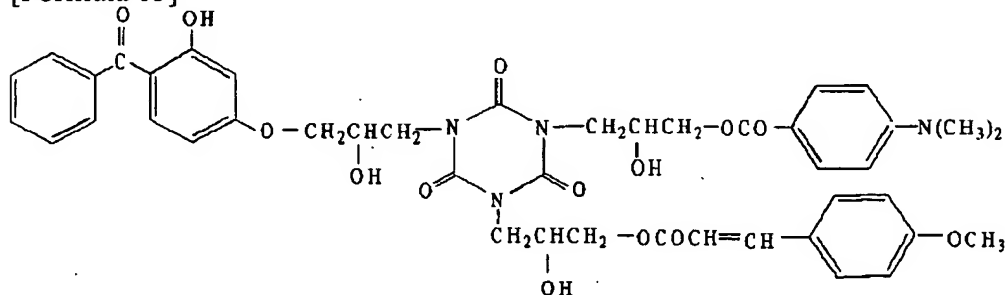
[0021]

[Formula 12]



[0022]

[Formula 13]



[0023]

[Example] Hereafter, an example explains the compound of this invention.

Xylenesg [ 50.0 ] and 2 and 4-dihydroxy benzophenone (product made from common chemical) 15.0g (0.07 mols) and tris (epoxy propyl) isocyanurate (product made from Nissan chemistry) 21.5g (0.07 mols) were taught to 300ml \*\* flask of composition of the [example[ 1 ]]-izing 8, and it agitated at 140 degrees C for 2 hours. 12.5g (Wako Pure Chem) (0.07 mols) of PARAMETOKISHI cinnamic acid was added here, 2-(2', 4'-dihydroxy phenyl)-5-chlorobenzo triazole (product made from common chemical) 18.3g (0.07 mols) was added after 2 more hour churning, and heating churning was carried out for further 5 hours. Then, the bottom xylene of reduced pressure was distilled out and light-yellowish-brown-color transparence liquid was obtained. 9:1 mixed solvents of isopropyl alcohol and an acetone were added to this, and it cooled to the room temperature, and the deposit crystal was filtered, it dried, and 64g (yield: 96.2% of the theory) of light-yellowish-brown-color fine particles with a melting point of 93-97 degrees C was obtained. The ultraviolet-region absorption field showed 250nm - 470nm.

[0024] It is 4-amino instead of the PARAMETOKISHI cinnamic acid used in the synthetic example 1 of the [example[ 2 ]]-izing 9. - 2, 2, 6, and 6-tetramethylpiperidine (product made from Huls) 11g (0.07 mols) was used, the same actuation as an example 1 was performed, and 62g (yield: 95.4% of the theory) of yellowish-brown-color fine particles with a melting point of 111-115 degrees C was obtained. The ultraviolet-region absorption field showed 250nm - 475nm.

[0025] 11.6g (Wako Pure Chem) (0.07 mols) of PARAJI methylamino benzoic acids was used instead of the 2-(2', 4'-dihydroxy phenyl)-5-chlorobenzo triazole of the synthetic example 1 of the [example[ 3 ]]-izing 13, the same actuation as an example 1 was performed, and 53.8g (yield: 90.0% of the theory) of light brown fine particles with a melting point of 79-84 degrees C was obtained. The ultraviolet-region absorption field showed 250nm - 360nm.

[0026]

[Effect of the Invention] This inventions are the absorbent of broader-based ultraviolet rays, and a thing which reaches or offers an ultraviolet ray absorbent for super-thin films like a paint film.

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[Translation done.]